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PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

Method of and Apparatus for the Production of Silicone Products

We, WERNER & PFLEIDERER, a Kommanditgesellschaft, organized and existing under the laws of the Federal Republic of Germany of 10 Theodorstrasse, 7000 Stuttgart-Feuerbach, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process and apparatus for the preparation of silicone products e.g. silicone oils, silicone greases, and silicone rubbers, from a mixture of starting materials.

To enable such silicone products to have particular properties, such as for example heat-resistance and resilience, temperatures reached during preparation must be maintained within a predetermined limit, and degasification and therefore the condensation of the individual components, must likewise be limited to a predetermined degree. In practice such control has been expensive to carry out, and is generally incomplete in effect.

By the practice of the invention, silicone products may be continuously produced with precisely predetermined properties.

The invention consists of a process for the preparation of silicone products from a mixture of starting materials, comprising continuously introducing the starting materials separately into a mixing chamber; mixing the said individual starting materials in the said mixing chamber; forcing the mixture thereby produced into a reaction chamber and conveying the said mixture through the said reaction chamber to a discharge member, the said reaction chamber having at least one degasification zone along its length whereby gases may be removed from the reacting mixture.

[Price 4s. 6d.]

According to a preferred and important feature of the invention, the speed at which the mixture passes through the reaction chamber is varied.

According to a further feature of the invention, additives, for example a filler and/or a deactivising agent, can be continuously fed to the mixture in the reaction chamber before it emerges from the reaction chamber, the temperature being constant. Alternatively, the reaction mixture may be heated while in the reaction chamber. Either method ensures that a point adjacent and upstream of the discharge member, the deactivating agent and/or heat stops a condensation reaction in the reaction mixture.

The reaction mixture is preferably conveyed through the reaction chamber by a screw conveyor.

The aforementioned steps ensure that the individual proportions of the starting materials are continuously fed in constant quantities and in a predetermined ratio, and that a relatively high vacuum can be applied in one or more stages inside the reaction chamber, depending on the required properties of the end product, thus reducing to the minimum the time spent by the mixture inside the reaction chamber. Since the speed at which the mixture passes through the reaction chamber can be varied in a very simple manner for instance as hereinafter described, the required properties of the emerging mixture, such as viscosity, resilience and the like, can be definitely determined merely by the speed at which the mixture passes through the reaction chamber.

The process according to the invention ensures reliable control and enables the required properties of the end product to be determined rapidly and with certainty, either by the metered feed of the activising

agent and/or heat, or by the speed at which the mixture passes through the reaction chamber and the immediately adjoining outlet member.

Apparatus for carrying out the invention comprises a mixing chamber; means for introducing starting materials separately in the said mixing chamber; a reaction chamber having at least two co-operating conveying members for the reacting mixture therein, conduit means for passing a mixture from the said mixing chamber to the said reaction chamber; discharge means for discharging reaction mixture from the said reaction chamber after conveyance therethrough; and at least one vacuum degasification point situated on the length of the reaction chamber.

For the performance of the method according to the invention, the starting materials are mixed in a screw extruder, the mixture being introduced into a reaction chamber having at least two co-operating conveying members, and the mixture continuously discharged therefrom through a discharge head connected to the reaction chamber. Conveniently the conveying members inside the reaction chamber are constructed in the form of self-cleaning screws which are mounted floatably inside the casing. This step ensures that no undesirable residues are left at any place in the unreaction chamber which might lead to uncontrollable reactions and therefore have an adverse effect on the required reaction controlled for the whole distance over which the mixture passes, and the resulting reliable determination of the properties of the end product.

The reaction chamber may comprise a number of assemblable casing members, one or more of which can be formed with introduction apertures for the addition of additives. Clearly, one or more of the casing members can also be formed with removal apertures for the application of the aforementioned vacuum. Conveniently, the casing portions are heated individually or in groups each comprising a number of casing members, thus ensuring controlle dtemperatures over the whole of the treating zone.

Embodiments of the invention are hereinafter described and illustrated in the accompanying drawings, of which.

Figure 1 is a diagrammatic general representation of apparatus for the preparation of silicone products according to the invention;

Figure 2 is a diagrammatic representation, partly in section, of apparatus for the preparation of silicone products according to the invention;

Figure 3 is a section taken along the line III-III of Figure 2;

Figure 4 is a longitudinal section of a portion of a discharge member adjoining the reaction chamber, suitable for discharge of silicone rubber;

Figure 5 is a longitudinal section of a portion of a discharge member adjoining the reaction chamber, suitable for discharge of a silicone grease, and

Figure 6 is a longitudinal section of a portion of a discharge member adjoining the reaction chamber, suitable for discharge of silicone oil.

Referring to Figure 1, components A, B, C of a mixture to be reacted to form a silicon product, are fed from individual reservoirs 1, 2 and 3 via metering members 4, for instance metering pumps, and lines 5, 6 and 7 connected thereto, to a mixer constructed in the form of a rapidly operating screw extruder 8. The individual components A, B and C are intermixed to form a mixture which is continuously forced via a line 9 to a reaction chamber 10 forming part of a screw extruder 11 whose construction is shown diagrammatically in greater detail in Figure 2. After it enters the screw extruder 11, the mixture is forced through the reaction chamber 10, a vacuum of, for instance 10 torr, being applied at places spaced out from one another via lines 12, 13, to degasify the mixture. Depending on the time taken by the mixture to pass through the reaction chamber 10, a very considerable degasification may take place as at the first degasification zone, so that if required a different vacuum can be applied via individual lines 12, 13.

In the zone of the discharge member 14 adjoining the reaction chamber 10 of the screw extruder 11, the required properties of the mixture are determined by deactivating agent being fed, and excess catalyst therefore decomposed, from a reservoir 15 via a conveying member 16, for instance in the form of a pump or screw means, and an adjoining line 17 which discharges into the end portion of the reaction chamber 10. The result is a forced brake-off of condensation. If required, plasticising agent can be added to the mixture from a reservoir 18 via a further conveying member 19 and a line 20, or fillers can be added via a further line 21, to determine the colour or other properties of the mixture. Various possible forms of the discharge member are illustrated in Figures 4, 5 and 6. Other variants of said embodiments may be used, depending on the particular product or circumstances.

Figures 2 and 3 show in greater detail the construction of the screw extruder 11. Instead of the metering members 4 taking the form of pumps, as shown in Figure 1, in Figure 2 gear pumps 4' feed the individual components A, B, C from the reser-

voirs 1, 2 and 3 to the mixer taking the form of a screw extruder 8, in which the components are mixed together and fed via a line 9 to reaction chamber 10 of screw extruder 11. The reaction chamber 10 is formed by individual placing sections 10′ which enclose the continuous screws 22, 23 (shown in Figure 3) over their whole length. The screws 22, 23 are disposed floatably inside the reaction chamber 10 and the individual casing sections 10′ and are driven in known manner by a drive motor 24, if necessary via interposed transmission members 25.

Disposed on the screws 22, 23 between those zones of the reaction chamber 10 in which the mixture is subjected to vacuum, are washers 26 which enable a varying vacuum to be applied at individual vacuum stages 27, 28 and 29. The number of vacuum stages can differ in dependence upon the material to be treated. The same thing also applies to the arrangement of the washers 26 on the screws 22, 23.

Since the reaction chamber 10 is formed of individual interconnected casing sections 10′, in practice the size of a suitable screw extruder can be determined in a very simple manner by matching the number of the casing sections with the length of the screws 22, 23, therefore enabling the machine to be increased or reduced in size. This can be achieved if, for instance as shown in Figure 2, the last vacuum stage 29 is uncoupled from the associated screw section, and the outlet member 14 is connected directly to the vacuum stage 28. Deactivating agents can be introduced via screw means 19′ and a line 20′ into the mixture before it is removed from the discharge member 14, in the zone of the last reaction portion or of the discharge member 14, as in the embodiment shown in Figure 2. As already stated, the construction of the discharge member 14 mainly depends on the nature and properties of the particular mixture, which according to the invention depends exclusively on the speed at which the mixture passes through the reaction chamber and/or the quantity of the deactivating agent fed or amount of heat added. Referring to Figure 4, a conically tapering connection member 30 is screwed to the end of the discharge member 14, the outlet aperture of the member 30 being closed by a plate 31 formed with individual bores 32. This kind of discharge head is more particularly suitable for silicone rubber the number and size of the outlet apertures 32 being selected, depending on the properties of the silicone rubber.

Instead of the closure plate 31, as shown in Figure 4, a line 33 as shown in Fig. 5 can be connected which can then be used for silicone greases. The outlet ends of the screws 22, 23 then force the finished silicone grease into containers provided for the purpose.

If however silicone oil is being prepared, a line 34 as shown in Fig. 6 extending to a delivery pump is connected to the tapering connection member 30, so that the pump, for instance a gear pump 35, pumps the silicone oil into containers provided for this purpose.

Alternatively, the arrangement can be such that the closure plate 31, the tubular member 33, or a line 34 connected to a pump 35 can be connected at choice to the tapering connection member 30.

The speed at which the mixture passes through the reaction chamber can be determined in a very simple manner by changing the speed of the driving motor or transmission thus altering the conveying speed of the screws 22, 23. In a very simple manner screw means can be provided at the required places in the reaction chamber for the addition of additives, thus ensuring reliable control of the whole reaction of the mixture and its required final properties.

The apparatus may be varied to control the temperatures inside individual casing sections 10′, and the degasification by means of individual vacuum stages 27, 28 and 29, and the feeding of the individual starting materials to the mixture, and also to the construction of the mixer in the form of a single or multiple-shaft screw extruder 8.

WHAT WE CLAIM IS:—

1. A process for the preparation of silicone products from a mixture of starting materials, comprising continuously introducing the starting materials separately into a mixing chamber; mixing the said individual starting materials in the said mixing chamber; forcing the mixture thereby produced into a reaction chamber and conveying the said mixture through the said reaction chamber to a discharge member, the said reaction chamber having at least one degasification zone along its length whereby gases may be removed from the reacting mixture.

2. A process according to Claim 1, in which the speed at which the mixture passes through the reaction chamber is varied.

3. A process according to Claim 1 or Claim 2, in which one or more additives are continuously added to the mixture at a constant temperature in the said reaction chamber.

4. A process according to Claim 3, in which at least one additive consisting of a filler and a deactivating agent is added to the said reaction mixture in the reaction chamber.

5. A process according to any of Claims 1 to 4, in which the mixture is heated in the said reaction chamber.

6. A process according to any of Claims 1 to 5, in which the said reaction mixture is conveyed through the said reaction chamber by a conveying screw.

7. A process for the preparation of silicone products as claimed in Claim 1, substantially as hereinbefore described.

8. Silicone products prepared by a process claimed in any of Claims 1 to 7.

9. Apparatus for the continuous preparation of silicone products, comprising a mixing chamber; means for introducing starting materials separately in the said mixing chamber; a reaction chamber having at least two co-operating conveying members for the reacting mixture therein, conduit means for passing a mixture from the said mixing chamber to the said reaction chamber; discharge means for discharging reaction mixture from the said reaction chamber after conveyance therethrough; and at least one vacuum degasification point situated on the length of the reaction chamber.

10. Apparatus according to Claim 9, in which the conveyor members constructed are conveyor screws disposed floatably inside a casing of the reaction chamber.

11. Apparatus according to Claim 9 or Claim 10, in which the reaction chamber comprises a plurality of assemblable casing members at least one of which is provided with inlet means for the introduction of an additive to reaction mixture in the reaction chamber.

12. Apparatus according to any of Claims 9 to 11, in which the said casing members are adapted to be heated individually or in groups.

13. Apparatus for the continuous preparation of silicone products, substantially as hereinbefore described and illustrated in any of the Figures of the accompanying drawings.

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4 SHEETS

COMPLETE SPECIFICATION

This drawing is a reproduction of the Original on a reduced scale.
SHEET 1

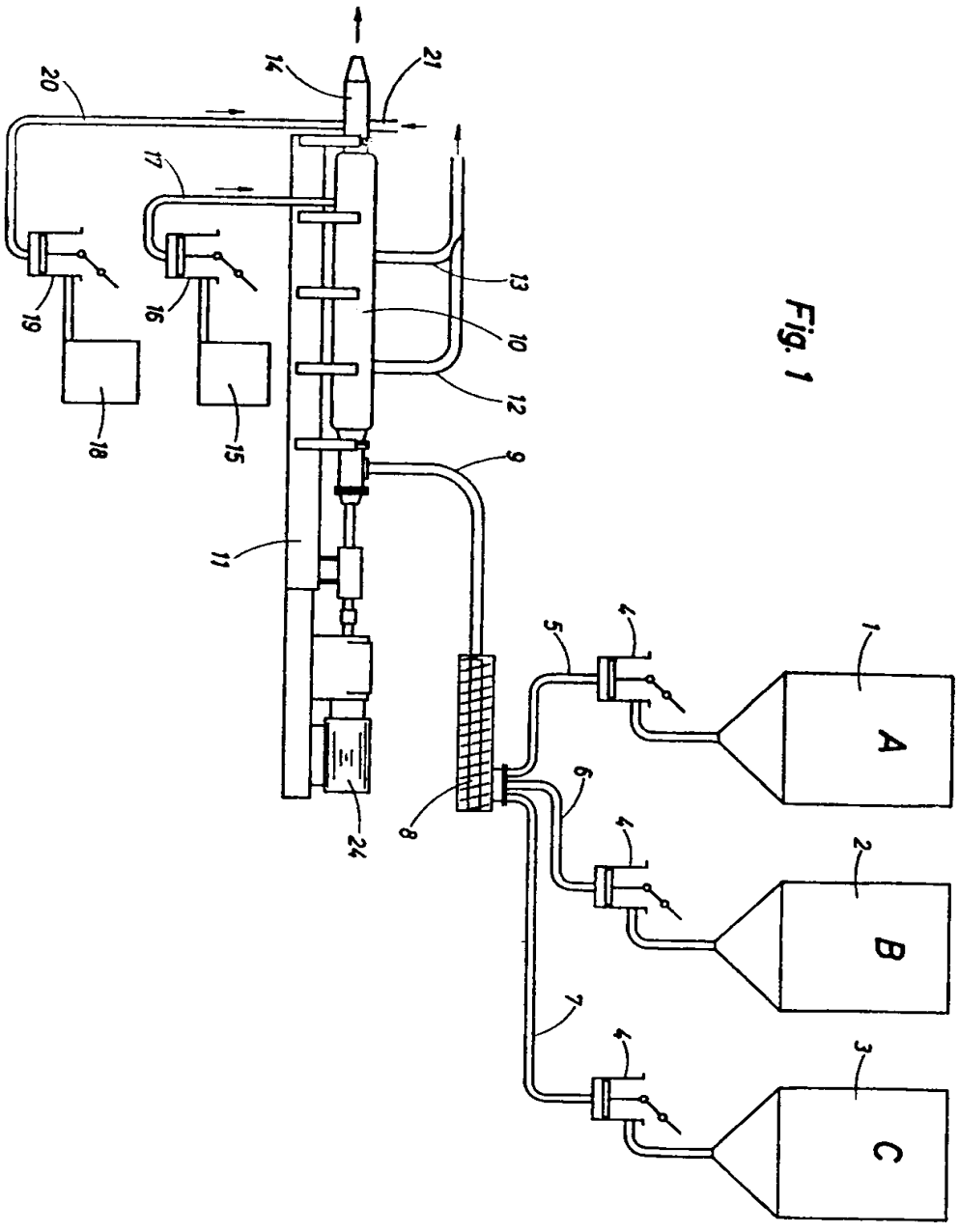


Fig. 1

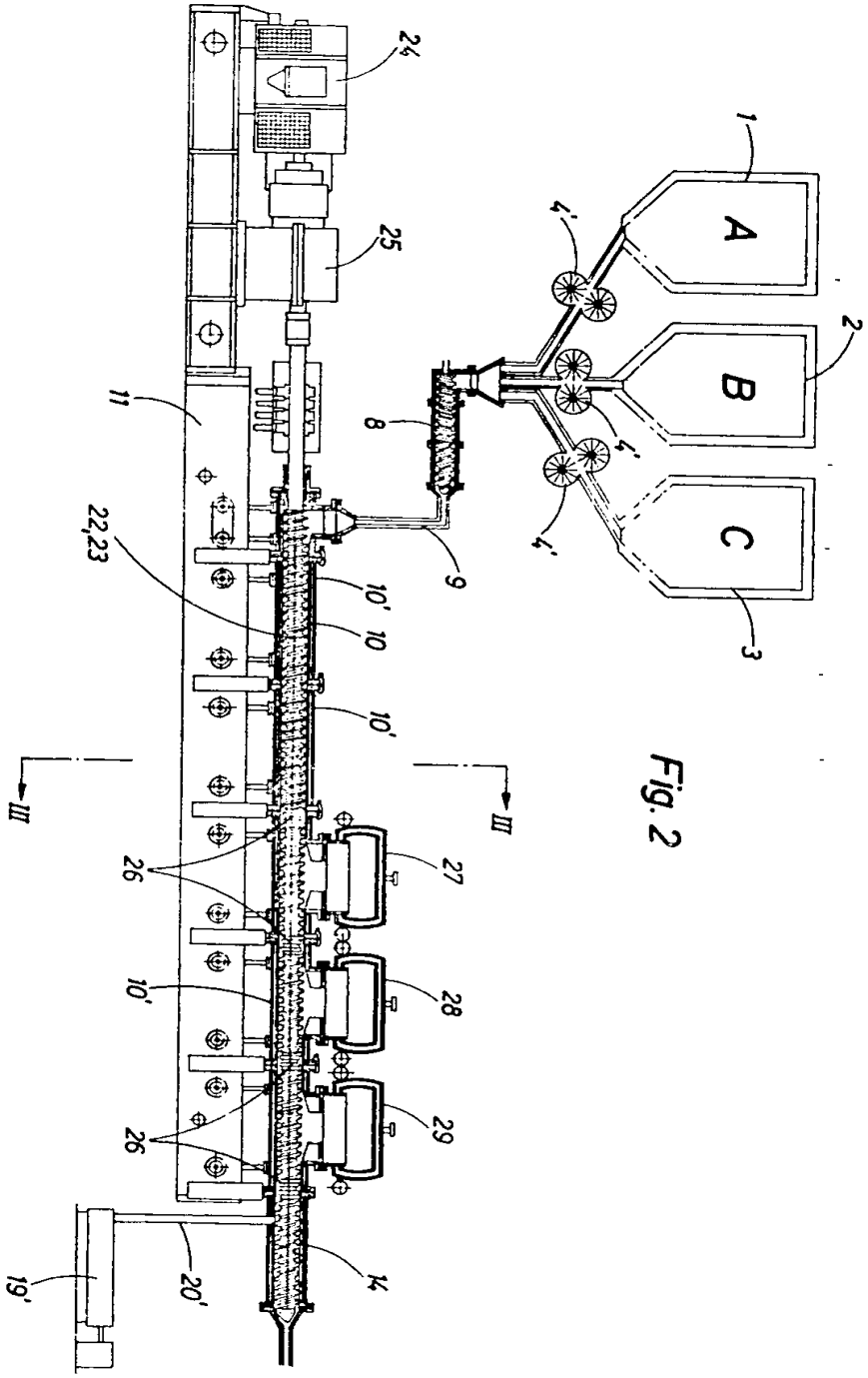
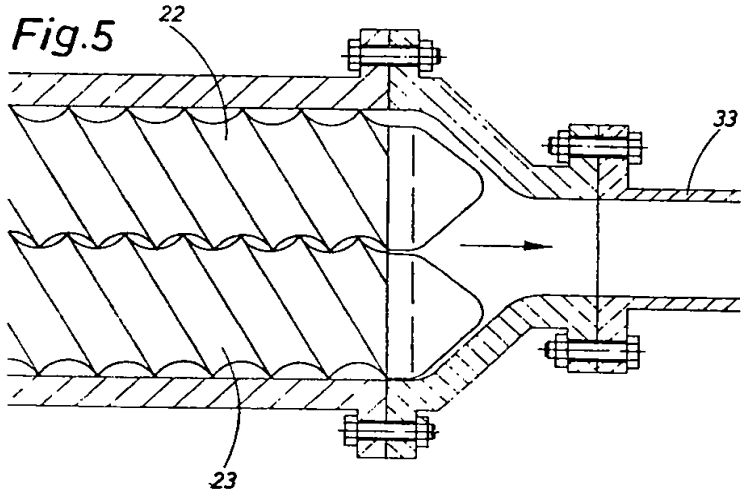


Fig. 2

Fig.5



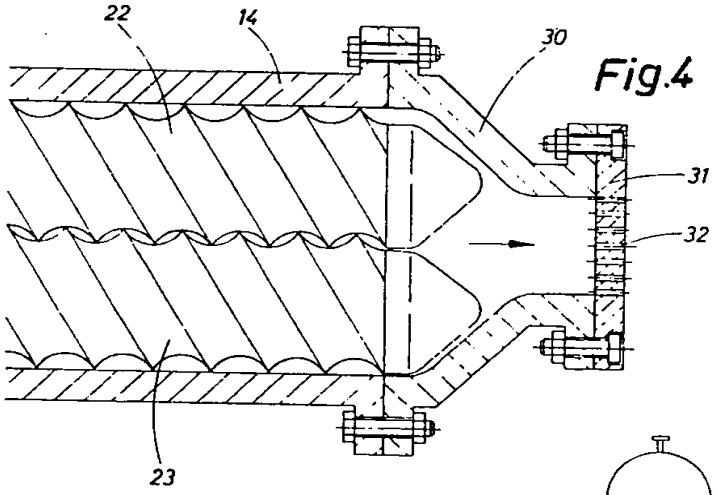
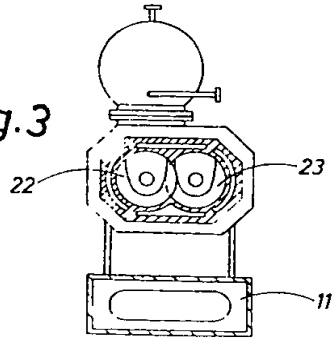


Fig.4

Fig.3



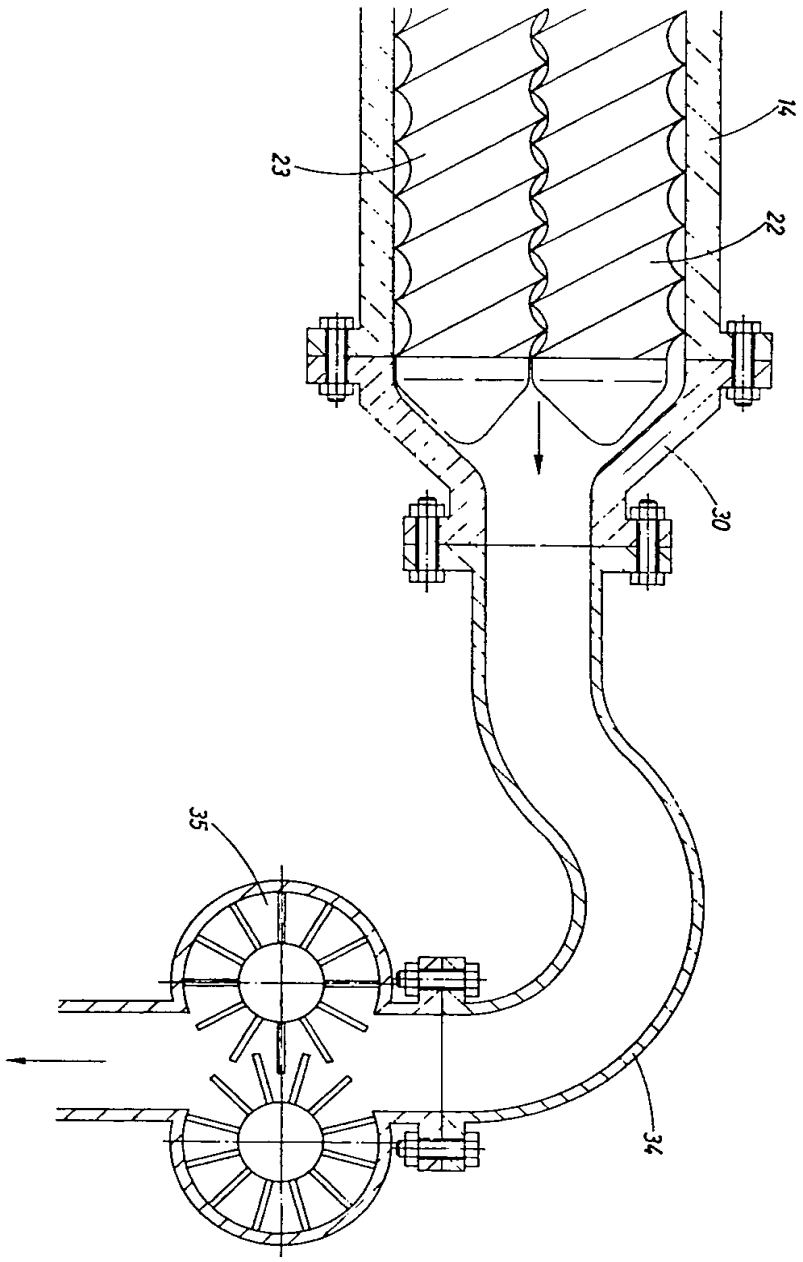


Fig. 6